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   Patents ADP number *(if you know it)* — 5808068001

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4. **Title of the invention** — VAPOUR DEPOSITION

5. **Name of your agent** *(if you have one)* — D YOUNG & CO

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Novel Processing Technique: Electrostatic and Flame Assited Vapour Deposition

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ABSTRACT

A novel and cost-effective vapour synthesis technique called *Electrostatic and flame assisted vapour deposition (EFAVD)* has been developed to deposit ceramic films and coatings in an open atmosphere. This technique combines the advantages of flame synthesis (FS), electrostatic spray deposition, chemical vapour deposition (CVD) and sol-gel techniques. In addition, this method can also be adapted for the production of ultrafine powders and near shape engineering parts.

INTRODUCTION

The applications of ceramics as structural coatings and functional electronic films are widespread and expanding rapidly. There are needs for improved methods of fabrication for ceramic films and coatings. Various conventional and commercial techniques such as CVD, physical vapour deposition (PVD), flame spraying deposition (FSD), combustion chemical vapour deposition (CCVD), and sol-gel have been investigated.

Both CVD and PVD involve the use of sophisticated and expensive deposition chamber and/or vacuum system. Applications of CVD/PVD methods to ceramic films are limited to the coating process in which the required film thickness and coating area are relatively small. In addition, CVD often leads to difficulties in controlling the stoichiometry of multicomponent oxide films and problem arise due to differences in vapour pressure of CVD reagents and low growth rate of deposited films. PVD method such as the radio frequency sputtering gives low deposition rates and low yield. Reactive magnetic sputtering and ion-beam sputtering both need expensive equipment and highly skilled technician. Conventional evaporation method has difficulties in depositing materials with high melting/vaporising temperatures, except the very expensive electron beam evaporation (EB-PVD) method. Moreover, EB-PVD has difficulties in controlling the stoichiometry of multicomponent oxide films.

FSD [1] produces films with morphology, microstructure and electrical properties dependent on temperature of substrate, coating concentration, carrier gas flow etc. It is therefore complex to control the variables and difficult to implement.

CCVD [2] has a low deposition rate and experiences difficulties in depositing dense thick film (>10 microns). Moreover, it involves the use of high flame temperature (>800°C), cause premature decomposition and the loss of some precursors to the surrounding.

For the production of ceramic film, sol-gel has two problems; the films tend to crack and the film has to have a definite thickness. These are major obstacles to utilization.

Similarly, there is a lack of processing methods that can produce ultrafine powders cost effectively. Materials with nanostructured have been recognised to exhibit attractive mechanical, electrical, optical and magnetic properties which are not observed in materials with coarse-grained structures. Nanostructured powders can be synthesized by chemical, physical and mechanical methods. Gas condensation in vacuum is the most applicable method for the synthesis of high purity ceramic nanopowders with well controlled particle size and distribution. However, such processing method is very expensive. Thus, limit the applications of ultrafine powders in high value added components.

Electrostatic and flame assisted vapour deposition (EFAVD) of films and coatings

EFAVD is a novel and cost-effective coating method which involves spraying atomised precursor droplets into a flame, within an electric field between the source and the substrate, where the charged aerosol undergo combustion and/or chemical reactions in the vapour phase near the vicinity of the substrate. This results in the formation of a stable solid film with excellent adhesion onto a substrate.

The electric field will charge the droplets and electrostatically attract the droplets from the outlet towards the substrate. In addition, an electric field and/or magnetic field could be created across the flame axis to steer and/or focus the sream of fine droplets in transit from the outlet to the substrate.

This technique combines the features and advantages of flame synthesis (FS), elelctrostatic assisted vapour deposition [3], chemical vapour deposition (CVD) and sol-gel techniques.

Key advantages of EFAVD include:

- simple/flexible/mobile equipment
- low cost, safe process using sol precursors and/or water based precursors
- relative low flame/deposition temperature for crystalline (e.g.550-800°C for Y_2O_3-ZrO_2)
- thin and thick film can be produced
- dense and porous film can be deposited onto dense or porous substrates. The deposition of dense film onto porous substrates involves the deposition of an amorphous interlayer).
- the choice of precursor is important. For the deposition of dense film , the precursor is based on sol precursor. The deposition of porous films can be based on sol or water soluble precursors.
- low consumption of precursor (e.g. 1 ml 0.05M to produce 1 micron thick film on 1cm x 1 cm substrate).
- high deposition rate
- substrate may be simple or complex in shape and may be ceramic or metal
- the process could be scale-up for large scale/large area production .
- single production process without the need for further heat treatment.
- simple, multicomponent oxides and doped oxides can be manufactured with well controlled structure in an open-atmosphere
- non oxides such as sulfides, carbides, etc. can be produced in a controlled atmosphere
- minimum loss of precursor to the surrounding as the electric field directs the charged aerosol precursors to the substrate/object forming table/powder collector
- by using sol precursor and incorporating gas condensation technique into the EFAVD, ultrafine

powders (in nanometer range) can be produced and collected in a cold collector (with a different polarity/potential than the source)

- by incorporating movable object forming table controlled by a computer aided design model (CAD) into the EFAVD, a 3-D object can be constructed layer by layer.

Process principle of the EFAVD for rapid prototyping

Atomised and charged precursor droplets are sprayed across an electric field in a flame source, where the charged aerosol undergo combustion and/or chemical reactions in the vapour phase near the object forming table to produce a 2D cross-sectional layer of the ceramic component. A dense 3D ceramic component is then constructed layer and layer with the required fine details. The movement of the table and hence the shape of the objet is controlled by the CAD.

It is also possible to move the flame source using a computer controlled CAD system.

Such direct rapid protoyping using flame source for the formation of the required material would be cheaper than those using laser source.

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[1] Choy, K. L., "Flame Assisted Vapour Deposition of Ceramic Films and Coatings", British Ceramic Proceedings, The Institute of Materials (1995) 65-74.

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[3] Choy, K. L., and Bai, W., " A method of depositiong Ceramic Films and Coatings", International PCT Application No. PCT/GB96/03105.

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